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**Interface Control and Mechanical  
Property Improvements in  
Silicon Carbide/Titanium Composites**

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Interface Control and Mechanical  
Property Improvements in  
Silicon Carbide/Titanium Composites

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## SUMMARY

Several composite systems made from titanium-matrix materials reinforced with silicon carbide fibers were investigated to determine composite-degradation mechanisms and to develop techniques to minimize loss of mechanical properties during fabrication and in service. Emphasis was on interface control by fiber or matrix coatings. Planar silicon carbide/titanium couples were made with thin aluminum or  $\text{Ti}_3\text{Al}$  interfacial layers to assess the effectiveness of the metallic layers in limiting silicon titanium reactions. Fibers and matrix materials were sputter-coated with aluminum, molybdenum, or vanadium to determine the effects of the coatings on basic fiber properties, fiber-matrix interactions, and composite properties. The effects of variations in fabrication temperature on composite properties were determined for composites consolidated by standard press-diffusion-bonding techniques.

X-ray diffraction analysis of the planar specimens showed that the formation of titanium silicides was greatly inhibited by the presence of the aluminum or the  $\text{Ti}_3\text{Al}$  layers. A 1- $\mu\text{m}$ -thick coating of aluminum improved the as-fabricated strength of silicon carbide fibers and reduced fiber degradation during fabrication. Applying an interfacial barrier by coating the matrix foils instead of the fibers was found to be an effective method for improving composite strength. Good-quality, well-consolidated composites were fabricated at temperatures well below those customarily used for silicon carbide/titanium composite fabrication.

## INTRODUCTION

Titanium-matrix composites have unique properties which make them attractive for a variety of aerospace applications for which high specific strength and stiffness at elevated temperatures are required. However, these composites have exhibited strengths well below those expected, based on fiber and matrix properties. (See ref. 1.) Deleterious fiber-matrix reactions during fabrication appear to be major contributors to the low strengths observed. It is well known that the fiber-matrix interactions play a major role in determining composite properties, and any reactions other than those required for good bonding are usually undesirable.

In general, there are three ways to minimize the effects of interface reactions: adjust fabrication parameters (time, temperature, and pressure) to reduce interactions; apply fiber or matrix coatings to prevent interactions (diffusion and reaction barriers); or modify fibers and/or matrix materials to tolerate interactions (sacrificial layers, new fibers or matrices, etc.). Successful application of either or all of these controls is expected to result in a material with improved properties.

Several composite systems made from titanium-matrix materials reinforced with silicon carbide fibers were investigated in this study to further the understanding of composite-degradation mechanisms and to develop techniques to minimize the degradation. Interfacial coatings were evaluated for their effectiveness in minimizing fiber-matrix reactions. Fibers and matrix materials were coated with various metals and then consolidated into composite panels by a press-diffusion-bonding procedure. The effects of the coatings on basic fiber properties, reaction-zone characteristics, and composite properties were determined. Standard press-diffusion-bonding procedures were used to fabricate composites at three different temperatures.

## MATERIALS

Three types of silicon carbide fibers were evaluated in this study. The simplest fiber, referred to as the SiC fiber, is beta silicon carbide with a nominal diameter of 142  $\mu\text{m}$ . (See ref. 1.) The strength of this fiber is relatively low with a wide range of variability. The second type of fiber, referred to as the SiC-C fiber, is the well-known silicon carbide fiber with a carbon-rich surface layer. (See ref. 1.) Except for the 1- $\mu\text{m}$ -thick outer carbon-rich layer, this fiber is the same as the SiC fiber. This outer layer was found to improve the handleability and strength of the fiber, presumably by filling in surface defects and thereby reducing the potential for undesirable stress concentrations. The SiC-C fiber is generally much stronger than the SiC fiber, but the strength also varies considerably.

The third fiber is designated SCS-2 and is essentially the SiC-C fiber with a silicon-enriched surface layer over the carbon-rich layer. (See ref. 2.) However, the outer surface layer remains silicon deficient relative to stoichiometric silicon carbide. The purpose of this surface layer is to improve the wettability of the fiber as well as to act as a sacrificial layer in titanium-composite consolidation. The surface layer reacts with the titanium and allows the carbon-rich layer (which gives the fiber its improved strength) to remain intact.

Two matrix materials were used in this study: TiA55 and Ti-6Al-4V. The TiA55 is a commercially pure titanium, readily available in foil form and relatively inexpensive. The Ti-6Al-4V, referred to as Ti(6-4), is the widely used titanium structural alloy.

Metal coatings investigated were aluminum, molybdenum, and vanadium. These materials were selected because they are common alloying elements for titanium and have been shown to inhibit titanium reactions with silicon carbide under some conditions. (See refs. 3 and 4.) Aluminum and  $\text{Ti}_3\text{Al}$  coatings were used in the X-ray diffraction studies because of the well-known aluminum rejection (or exchange reaction) mechanism observed in SiC/Ti systems. (See refs. 5 and 6.)

## EXPERIMENTAL AND ANALYTICAL PROCEDURES

### Planar Specimens

The formation of various titanium silicides can be detrimental to composite properties. (See refs. 6 and 7.) Therefore, studies were conducted to see if the migration and subsequent reactions of silicon and titanium in SiC/Ti systems could be minimized by a metallic barrier. Planar specimens were used instead of fibers or composites to simplify data analysis and interpretation. Reference specimens were made by sputtering 2  $\mu\text{m}$  of silicon carbide onto a 22-mm-diameter, 3-mm-thick pure-titanium disk. Other specimens were made by first sputtering 1  $\mu\text{m}$  of aluminum on titanium disks. Silicon carbide was then sputtered onto some of these aluminum-coated disks to form SiC/Al/Ti samples. The remaining aluminum-coated specimens were heat treated at 635°C for 1 hr to develop a  $\text{Ti}_3\text{Al}$  surface and then coated with SiC to form SiC/ $\text{Ti}_3\text{Al}$ /Ti samples. All samples were then exposed to temperatures typical of composite fabrication (875°C) and examined, by X-ray diffraction techniques, after various exposure times to determine the relative amounts of titanium silicide formed.

A conventional diffractometer with a high-intensity, fine-focus copper tube was used to generate the X-ray data. The diffractometer is equipped with a device that allows the specimen to be oscillated about one axis while being rotated about another

(perpendicular) axis. Oscillating and rotating the specimen help minimize the effects of large grains or preferred crystallographic orientation on the diffracted-beam X-ray intensity. X-ray reflections were recorded for all titanium silicides identifiable in each planar sample. Integrated intensities for the titanium silicides were then determined with an electronic planimeter. These integrated intensities were used as a measure of the relative amounts of titanium silicides formed in the samples and, hence, as a measure of the extent of the titanium-silicon reactions. The data from the SiC/Ti specimens were used as the reference. (See ref. 8 for full details of X-ray analysis procedures.)

### Fiber Studies

Fiber studies were conducted in which conditions at the fiber-matrix interface were approximated by sputtering titanium, or aluminum and then titanium, onto the fiber and exposing the fiber to temperatures and times typical of composite consolidation. The effects of these treatments on fiber properties were determined by conducting tensile tests on individual fibers before and after treatments. From 30 to 50 fibers of each type were tested. The data from these tests were used to generate histograms of fiber fracture strengths from which average strengths as well as strength distributions could be obtained for each combination of fiber, coating, and heat treatment.

To determine how the differences in fiber properties relate to differences in projected composite properties, a simple analysis (ref. 9) was used to calculate projected composite tensile strengths based on actual fiber-strength distributions. Because the tensile properties of all the fibers tested varied over wide ranges, a projected composite strength based on customary rule-of-mixtures analyses and average material properties did not seem appropriate. A rule-of-mixture calculation gives realistic composite properties only if individual fiber strengths do not differ significantly from the average strength. The analysis used in the present study takes into account the effects of individual fiber failures, matrix plasticity, and residual stresses due to differential thermal expansion of the fibers and the matrix. This analysis was used to predict composite strengths by using the measured properties of the treated and untreated fibers and published titanium-matrix properties. All calculations used herein were based on a fiber volume fraction of 0.30.

### Composite Panels

Uniaxially reinforced composite panels were fabricated with various combinations of matrix materials, fibers, and coatings to determine the effects of coatings on actual interface structure and composite properties. The TiA55 foils were sputter coated with either aluminum, molybdenum, or vanadium and then consolidated into composite panels with the SiC-C (ref. 10) or the SCS-2 fibers. Some of the aluminum-coated foils were heat treated prior to consolidation to develop a  $Ti_3Al$  layer. Panels were also made with uncoated Ti(6-4) foil and aluminum-coated SCS-2 fibers. Base-line panels were made without any fiber or matrix coatings or treatments. In addition, panels were made without any reinforcing fibers at all in order to determine the effects of the fabrication process on the matrix materials alone. Panels made with coated foils are referred to herein as coated panels, and panels made with uncoated foils are referred to as uncoated panels.

All panels were 10 cm by 10 cm by 4-ply thick and were consolidated by customary press-diffusion bonding. Fabrication temperatures from 760°C to 930°C and pressures

from 41 MPa to 138 MPa were used. Fabrication time for all panels was 30 min at the prescribed temperature. The panels were machined into 1.3-cm by 10-cm rectangular samples for tensile testing and metallurgical analysis. Composite tensile specimens were tested with the reinforcing fibers parallel to the longitudinal direction. All tensile tests were performed at room temperature. (See ref. 9 for details of test procedures.) Three to five specimens from each panel were tested. Polished cross sections and fracture surfaces of tested composites were examined by scanning electron microscopy (SEM) and electron microprobe analyses.

## RESULTS AND DISCUSSION

### Interfacial Barrier Effects

Planar specimens.— The effects of Al and  $\text{Ti}_3\text{Al}$  interfacial barriers on the formation of titanium silicides in the planar specimens are shown in figure 1. Relative (integrated) X-ray intensities of all identifiable titanium silicides are shown as a function of time at 875°C for each type of specimen. For heating times up to 1 hr, both the Al and  $\text{Ti}_3\text{Al}$  layers reduced silicide formation significantly, with the  $\text{Ti}_3\text{Al}$  being somewhat more effective.

Generally, the Al and the  $\text{Ti}_3\text{Al}$  impede bulk migration of titanium toward the SiC and, hence, inhibit the formation of titanium silicides. In addition, in the  $\text{Ti}_3\text{Al}$  system, the titanium is tied up in the relatively stable, high-melting-temperature aluminides and, thus, an activation energy barrier to the formation of silicides is introduced. These effects are further enhanced because the Al is rejected from the reaction zone (refs. 5 and 6) and the concentration of Al near the interface increases as the reactions proceed.

SiC-C/TiA55 composites.— To determine whether metallic coatings are as effective in reducing the fiber-matrix interactions in a composite material as the planar-specimen studies indicated they might be, TiA55 foils were coated with Al, Mo, or V and consolidated into composites with the SiC-C fiber. After fabrication, the coated foil panels were exposed to 875°C for 25 hr to develop thick reaction zones to facilitate measurements and comparisons. The effect of the coatings on reaction-zone thickness is shown in figure 2. The Mo and V coatings resulted in reaction-zone thicknesses slightly greater than those in the panel with no coating. However, the Al coating did significantly restrict reaction zone growth, as suggested by the planar-specimen studies. Photomicrographs of polished cross sections of SiC-C/TiA55 composites (fig. 3) showed the reaction-zone thickness in the as-fabricated Al-coated panel to be about one-third of that in the uncoated panel.

Although the SiC-C fiber is quite strong, composites fabricated with the fiber generally have poor strength, presumably because of high fiber-matrix reaction rates depleting the carbon-rich layer and degrading the fiber. It was presumed that reducing the amount of reaction would improve the strength of the fiber. However, in the present study, the reduction in reaction-zone thickness (fig. 3) did not result in a corresponding increase in composite strength for the SiC-C/TiA55 system. The same carbon-rich layer that gives the SiC-C fiber its improved strength appears to play a major role in producing poor composite properties.

For the uncoated panel, the high reaction rates caused the carbon-rich layer to be essentially consumed and resulted in a continuous, gradated interface region with relatively good fiber-matrix bonding even though the fiber may have been weakened. For this panel, photomicrographs of leached fibers (fig. 4(a)) showed significant



fiber breakup adjacent to composite fracture surfaces. This fiber breakup indicated that, because of the relatively good bonding, the matrix was able to transfer some of the loads away from the fracture surface. On the other hand, the small amounts of reaction in the coated panel (SiC-C/TiA55(Al)) left most of the carbon-rich layer undisturbed, which resulted in relatively poor bonding, as evidenced by essentially no fiber breakup near the composite fracture surface. (See fig. 4(b).) Because of these results, the SiC-C fiber was discounted as a viable candidate for high-strength titanium composites, and research emphasis was placed on the other fibers.

#### Coating Effects on Fiber Strength

Because the carbon-rich layer on the SiC-C fibers seemed to cause problems in the composites, fiber-coating studies were conducted to determine whether an aluminum coating could be used as a substitute for the carbon-rich layer to enhance fiber strength while also serving as an effective barrier to undesirable SiC-Ti reactions. The SiC fibers and SiC fibers coated with a 1- $\mu$ m-thick layer of aluminum were tested in the as-fabricated condition to determine the effects of the coating on fiber strength. Fiber-strength histograms are shown in figure 5. The data for the SiC fibers (fig. 5) showed a wide range in tensile strength (approximately 0.5 to 3 GPa), thus indicating a highly variable fiber. Coating the fiber with aluminum (denoted by SiC(Al)) reduced the variability considerably by eliminating the very low strength fibers. This behavior is significant because these low-strength fibers play a major role in determining the composite properties. Therefore, it appeared that the aluminum served in somewhat the same capacity as the carbon layer in improving fiber properties.

To determine whether this kind of improvement in fiber properties would be maintained in the presence of a titanium matrix, SiC fibers were coated with titanium, or aluminum and then titanium, exposed to composite fabrication temperatures and then tested for tensile property changes. Figure 6 shows the fiber-tensile-strength distributions. As seen previously, the as-received SiC fibers showed a wide range of fracture strengths including some very low strengths. As expected, depositing titanium directly onto the fiber and heat treating the fiber (SiC(Ti)) reduced the strength even further, thus showing the severe fiber degradation caused by the reaction with the titanium. However, the strengths of heat-treated, aluminum titanium coated fibers (SiC(Al)(Ti)) were about three times those of the SiC(Ti) fibers, again showing the effectiveness of aluminum in inhibiting reactions that cause fiber degradation. Similar tests were run for the recently developed SCS-2 fibers, and the strength distribution was also found to be fairly wide. However, the fiber was generally stronger than the SiC. Coating the SCS-2 fiber with aluminum and titanium and exposing it to composite-consolidation conditions had a minor effect on the fiber-strength distribution, and the fiber remained significantly stronger than the SiC. (See fig. 6.)

The analysis from reference 9 was used to calculate projected composite tensile strengths based on these measured fiber-strength distributions and TiA55 matrix properties. The results for 0.30-fiber-volume-fraction composites are shown in figure 7. The strength distribution for the aluminum-coated fiber resulted in a projected composite tensile strength (SiC(Al)/TiA55) almost 2 1/2 times that for the uncoated SiC fibers (SiC/TiA55). Even further increases in projected composite strength were obtained for the SCS-2 fiber system (SCS-2(Al)/TiA55). Because of the apparent superiority of the SCS-2 fiber, it was used for the composite-panel fabrication in the remainder of this study.

## SCS-2 Reinforced Composite Panels

The SCS-2 fiber was used to make composites for testing and analysis to determine whether the projected composite properties for this use this fiber with the  $\text{Ti}_3\text{Al}$  interfacial barrier could be realized in an actual composite. The TiA55 foils were coated with aluminum, heat treated to develop a stable  $\text{Ti}_3\text{Al}$  surface layer, and then consolidated into composite panels with SCS-2 fibers. Other panels were fabricated with no interfacial coating. Polished and etched cross sections of typical panels are shown in figure 8. The SEM and microprobe analysis showed that the  $\text{Ti}_3\text{Al}$  in the SCS-2/TiA55(Al) panel was uniformly distributed around the fibers and was confined to the fiber-matrix interface and to the regions where the coated foils joined together between fibers. These results show that applying the coating to the matrix is an effective technique for interface control.

Electron microprobe traces of the fiber-matrix interface regions in panels fabricated with coated and uncoated titanium foil (fig. 9) showed different interface structures. The figure shows elemental distributions in the area of the interfaces. In the uncoated panel, the titanium, silicon, and carbon interdiffused to form the generally detrimental silicide interface; whereas in the aluminum-coated panel, the carbon and silicon were, for the most part, contained within the original confines of the fiber by the  $\text{Ti}_3\text{Al}$  barrier. Thus, the potential for undesirable reactions was significantly reduced.

Polished cross sections and fracture surfaces of coated and uncoated panels are shown in figure 10. In contrast to the SiC-C fiber (fig. 4), the SCS-2 fiber-matrix bonding seemed to be better for the coated system than for the uncoated system. Polished cross sections of the uncoated system (fig. 10(a)) showed more fiber-matrix disbands, and the fracture surfaces showed somewhat more fiber pull-out than the coated system (fig. 10(b)).

Typical stress-strain data for the SCS-2/TiA55 system (fig. 11) showed that the coated panels had higher strengths and they also failed at higher strains. Although the initial tangent modulus (referred to herein as modulus) was about the same for each material, as the material strained the modulus of the coated panel remained constant out to relatively high stresses and strains, whereas the modulus of the uncoated panel decreased throughout the test.

A summary of the SCS-2/TiA55 tensile strengths is given in figure 12. The TiA55 bar in the figure shows the strength of an unreinforced panel which was consolidated under the same conditions as the reinforced panels at 816°C. The strength of the unreinforced panel was about the same as that of as-received titanium sheet material, which indicates that the composite processing did not significantly affect the matrix material itself. Coating the TiA55 foil with aluminum significantly improved the composite strength (from 0.8 to 1.2 GPa) for a given fabrication temperature, in this case 816°C. Reducing the fabrication temperature from 816°C to 760°C appeared to have about the same effect on the composite strength. The 760°C temperature is well below the range of 870°C to 930°C which is customarily used to fabricate titanium composites, but this low temperature appeared to produce well-consolidated panels with good properties. However, fabrication of highly formed structural components or parts with complex shapes may require the higher temperatures, in which case the interfacial barrier would be important.

For the Ti(6-4) matrix studies, instead of coating the matrix the SCS-2 fibers were coated with aluminum and then consolidated into composites with the uncoated alloy foil. Tensile strengths for the SCS-2/Ti(6-4) systems are shown in fig-

ure 13. The strength of the unreinforced panel (Ti(6-4)) which was consolidated at 816°C was slightly greater than that of annealed Ti-6Al-4V sheet material, which again shows that the composite-fabrication conditions did not appreciably affect the matrix material. The SCS-2 reinforced panel (SCS-2/Ti(6-4)) fabricated at 927°C (typical fabrication temperature for reinforced titanium composites in ceramic-platen presses) was not any stronger than the unreinforced panel fabricated at 816°C. The high fabrication temperature caused extensive fiber-matrix reactions negating any benefit from the fibers. Panels fabricated with aluminum-coated fibers, however, were somewhat stronger than those with uncoated fibers. Again, a reduction of the fabrication temperature, in this case from 927°C to 816°C, also produced a significant improvement in composite strength.

### CONCLUSIONS

Several composite systems made from titanium matrix reinforced with silicon carbide fiber were investigated to determine composite degradation mechanisms and to develop techniques to minimize loss of mechanical properties during fabrication and in service. The conclusions from the investigation are as follows:

1. X-ray diffraction studies on planar samples showed that silicon titanium reactions in silicon carbide/titanium systems were reduced significantly by introducing aluminum or  $Ti_3Al$  layers into the SiC/Ti system. The  $Ti_3Al$  was somewhat more effective in reducing the reactions than pure aluminum.

2. Fiber studies showed that coating the fiber with a 1- $\mu m$ -thick layer of aluminum improved the as-fabricated strength of a stoichiometric SiC fiber and reduced the fiber degradation during exposure to composite-fabrication conditions. These improvements in fiber properties carried over into improved composite properties.

3. Applying an interfacial barrier by coating the matrix foils instead of the fibers was found to be an effective method for improving composite strength. Composites made with aluminum-coated TiA55 matrix foils had about 50 percent greater tensile strength than composites made with uncoated foils.

4. Significant improvements in composite strength were obtained by reducing the fabrication temperature. Strong, well-consolidated composites were fabricated at temperatures well below those customarily used for SiC/Ti composite fabrication.

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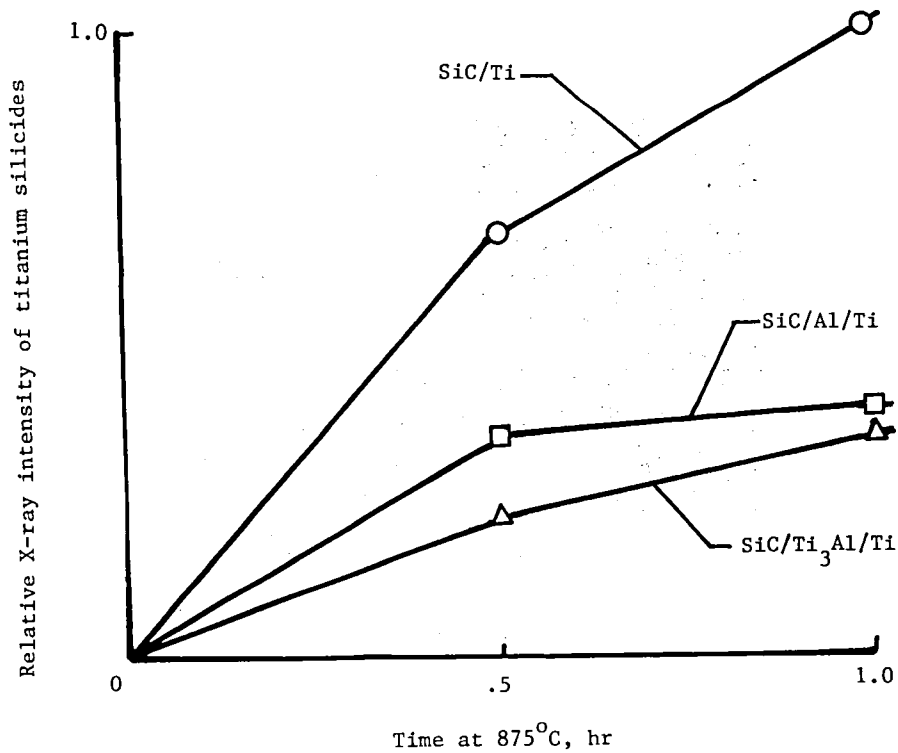


Figure 1.- Effect of metallic barriers on titanium silicide formation at 875°C. Planar specimens.

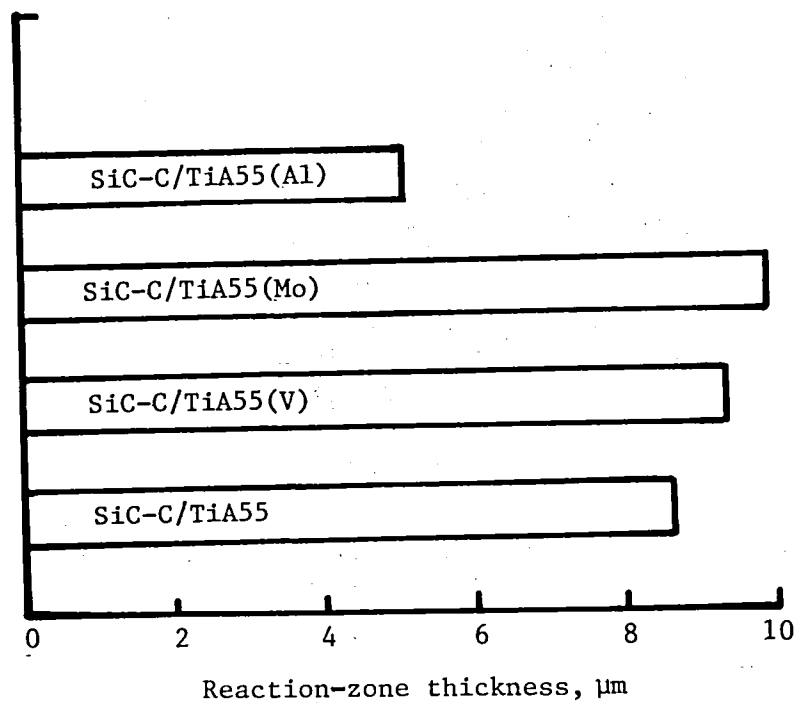
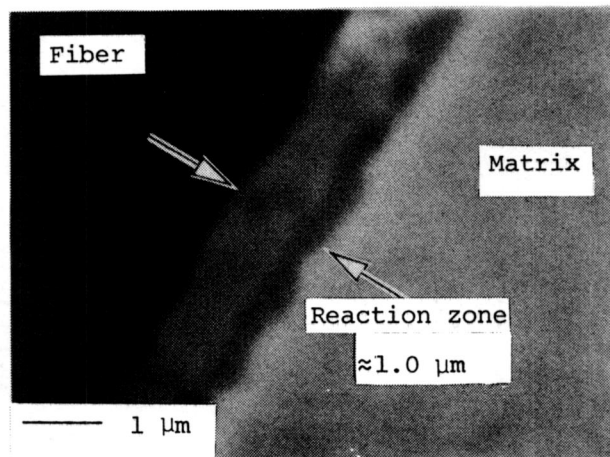
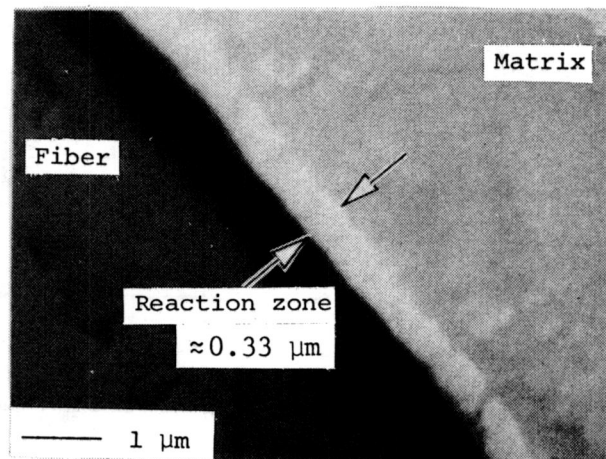


Figure 2.- Effect of metal coatings on reaction-zone thickness in SiC-C/TiA55 composites. Exposed at 875°C for 25 hr.



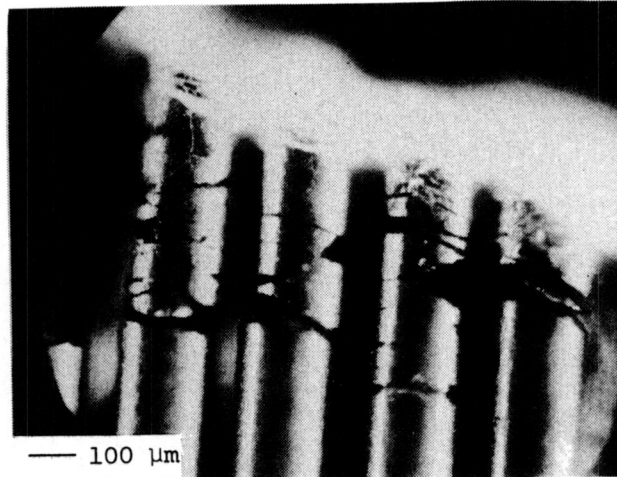
(a) SiC-C/TiA55.



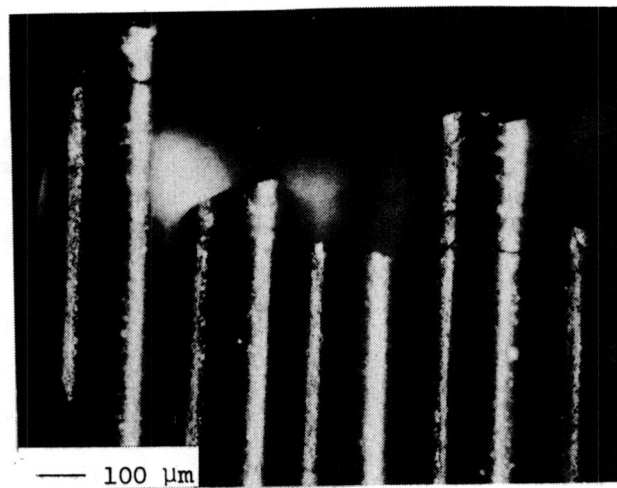
(b) SiC-C/TiA55(Al).

Figure 3.- Fiber-matrix interface in as-fabricated  
SiC-C/TiA55 composites.

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(a) SiC-C/TiA55.



(b) SiC-C/TiA55(Al).

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Figure 4.- Leached fibers near fracture surface of SiC-C/TiA55 composites. Fabricated at 875°C for 30 min at 103 MPa.

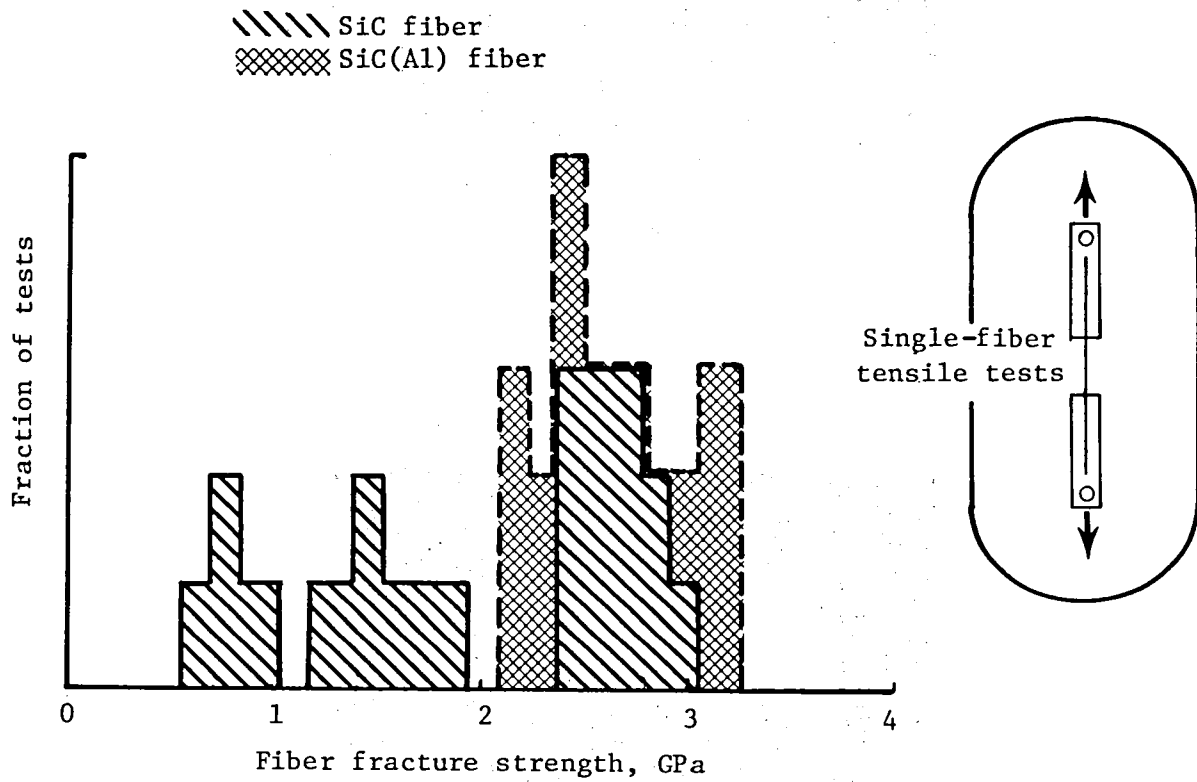


Figure 5.- Effect of aluminum coating on as-received SiC fiber strength.



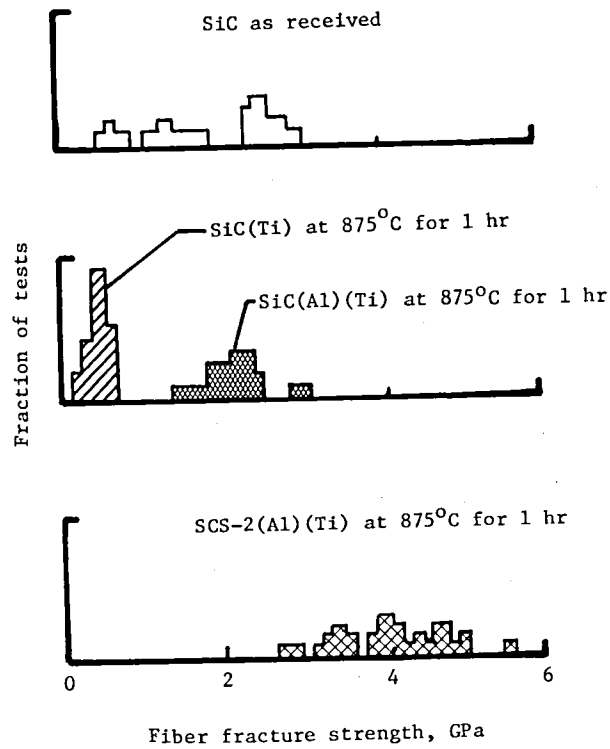


Figure 6.- Effect of coatings and heat treatment on fiber tensile strength.

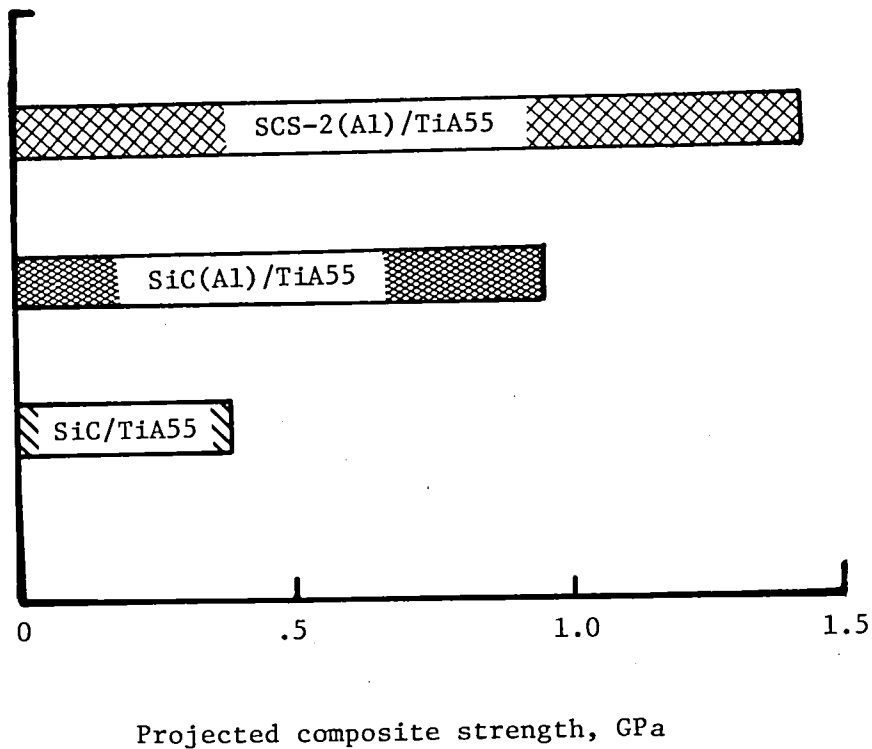
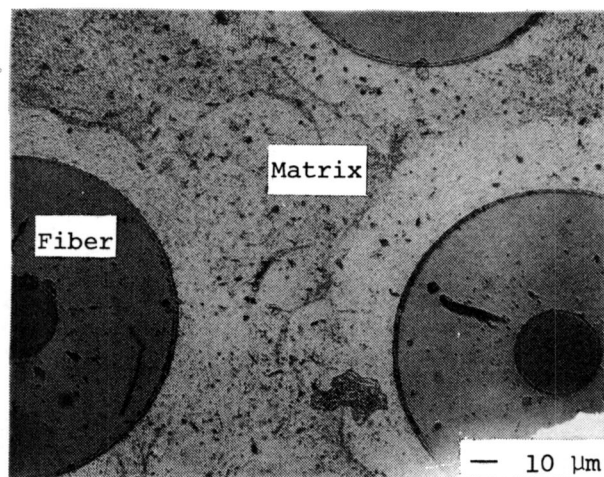
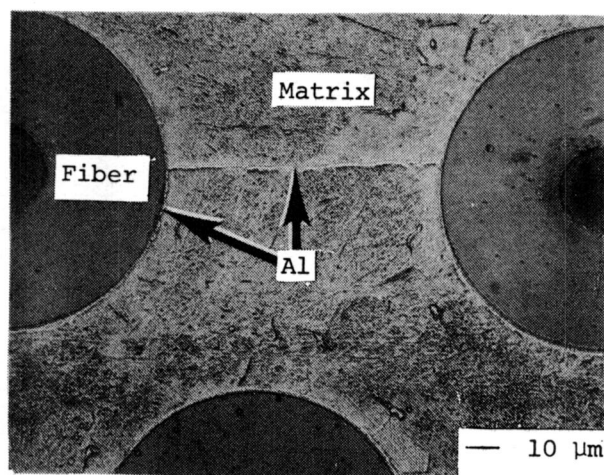


Figure 7.- Projected composite tensile strength for measured fiber-strength distributions. 0.30 fiber volume fraction.



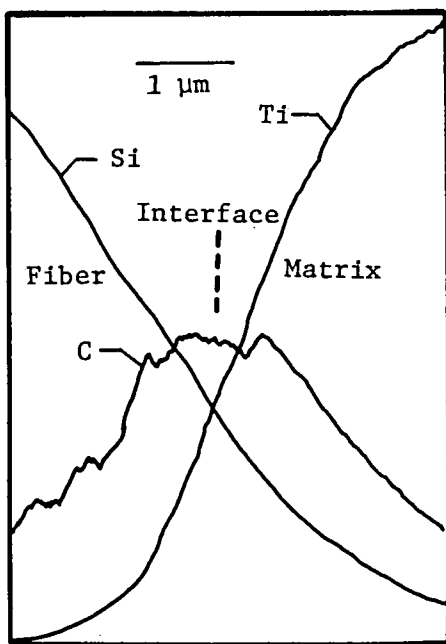
(a) SCS-2/TiA55.



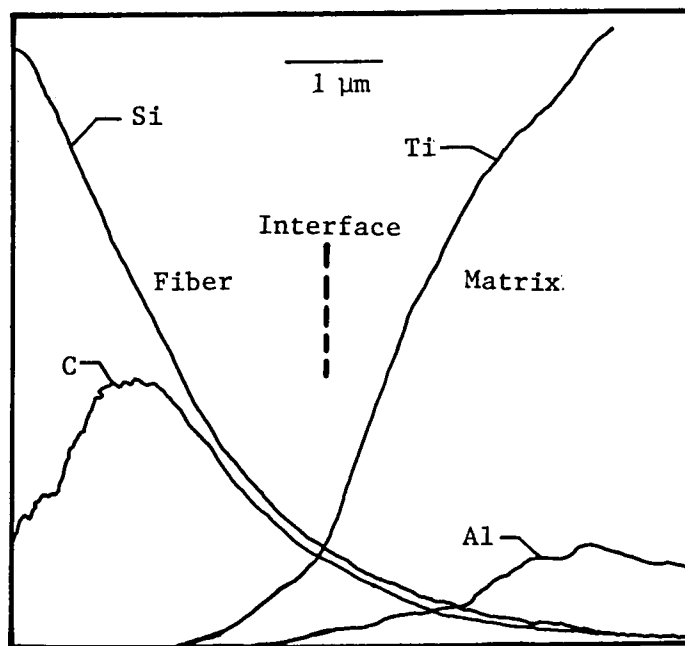
(b) SCS-2/TiA55(Al).

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Figure 8.- Polished cross sections of SCS-2/TiA55 composites.

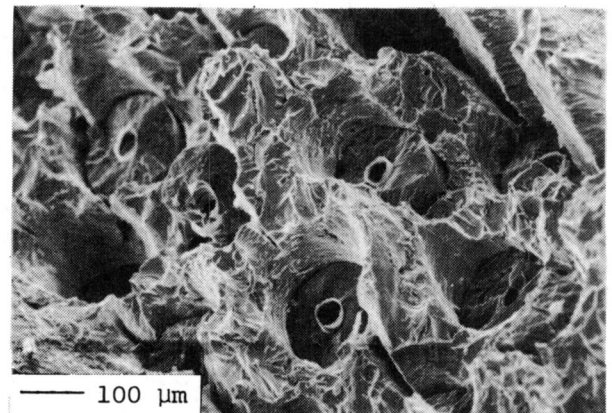
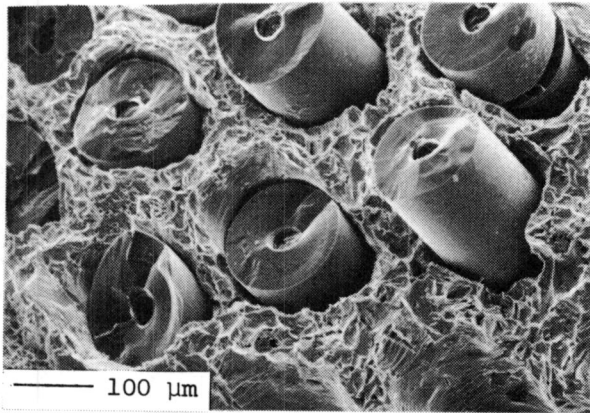
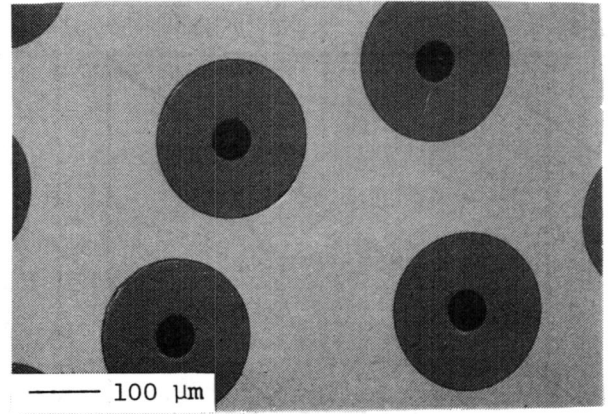
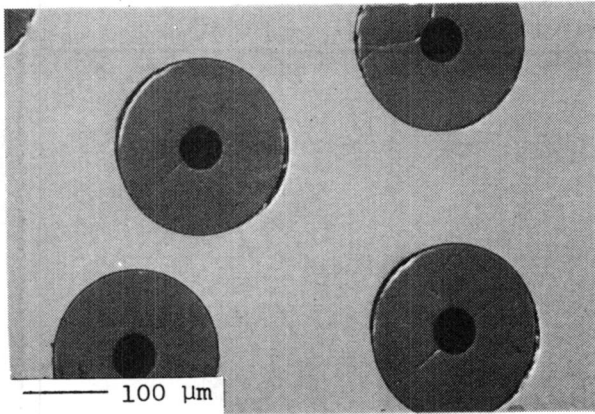


(a) SCS-2/TiA55.



(b) SCS-2/TiA55(Al).

Figure 9.- Elemental distributions across SCS-2/TiA55 interfaces.



(a) SCS-2/TiA55.

(b) SCS-2/TiA55(Al).

Figure 10.- Polished cross sections and fracture surfaces of SCS-2/TiA55 composites.

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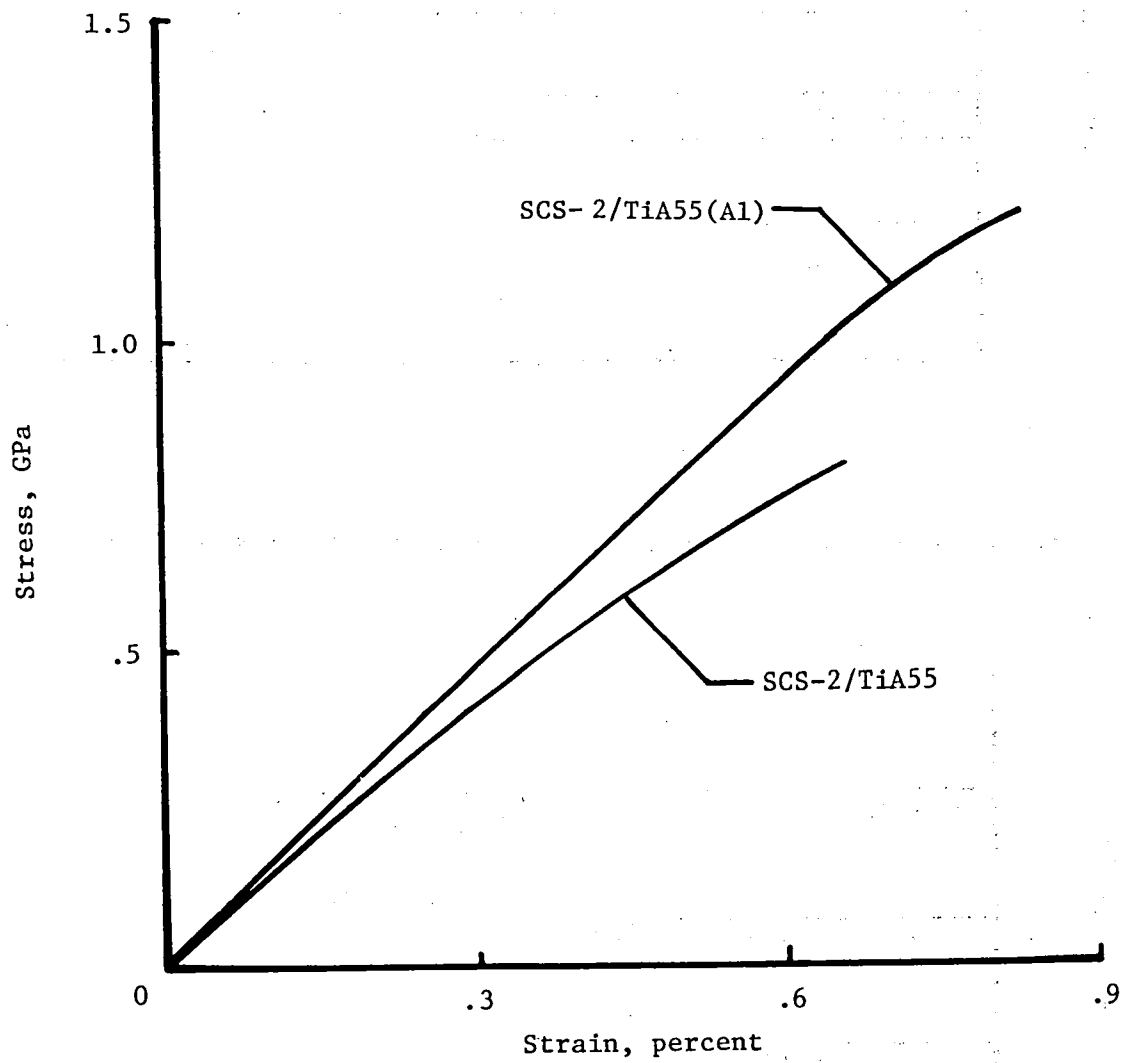


Figure 11.- Effect of matrix coating on tensile stress-strain behavior of SCS-2/TiA55 composites.

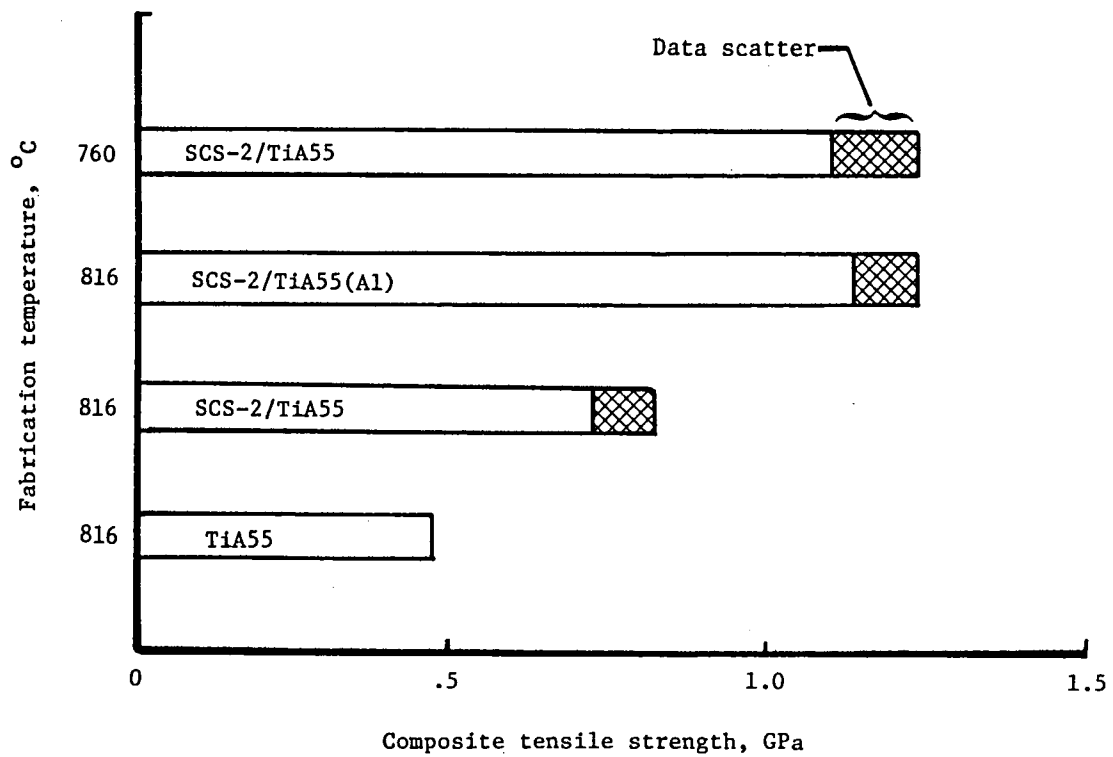


Figure 12.- Measured tensile strength of SCS-2/TiA55 composites.

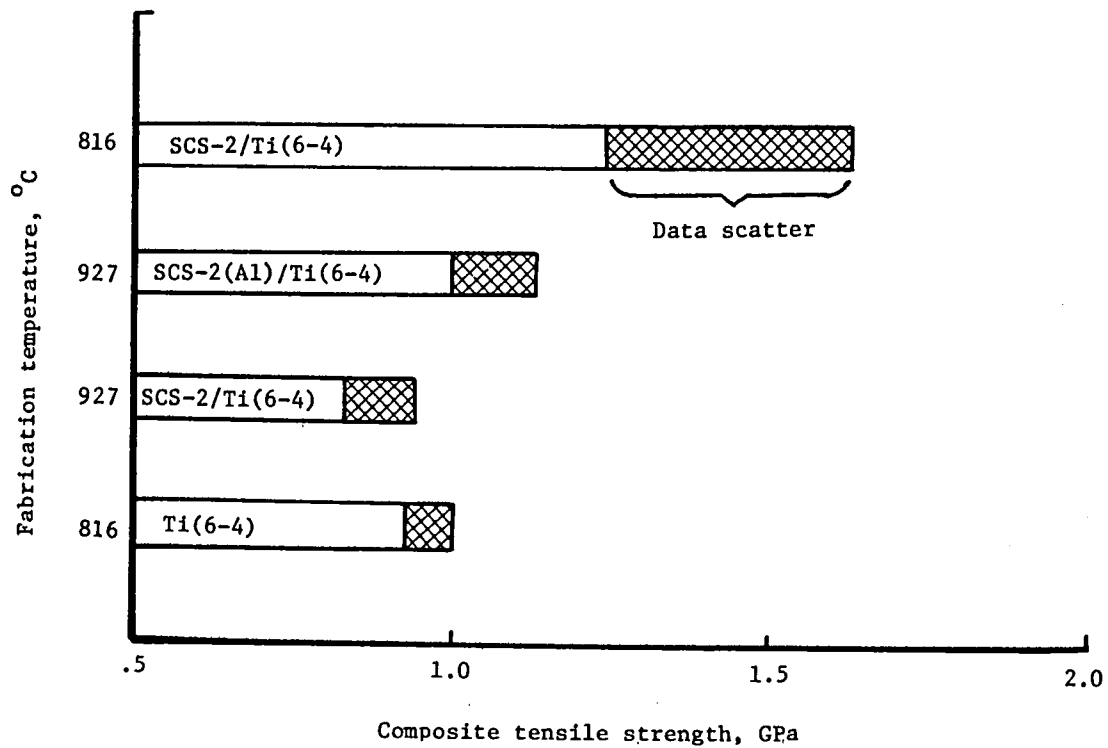


Figure 13.- Measured tensile strength of SCS-2/Ti(6-4) composites.









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16. Abstract  Several composite systems made of titanium matrix reinforced with silicon carbide fiber were investigated to obtain a better understanding of composite-degradation mechanisms and to develop techniques to minimize loss of mechanical properties during fabrication and in service. Emphasis was on interface control by fiber or matrix coatings. X-ray diffraction studies on planar samples showed that the formation of titanium silicides was greatly inhibited by the presence of aluminum or Ti <sub>3</sub> Al layers at the fiber-matrix interface, with the Ti <sub>3</sub> Al being more effective in reducing the reactions. Fiber studies showed that coating the fiber with a 1-μm-thick layer of aluminum improved the as-fabricated strength of a stoichiometric SiC fiber and reduced the fiber degradation during exposure to composite-fabrication conditions. Applying an interfacial barrier by coating the matrix foils instead of the fibers was found to be an effective method for improving composite strength. Reducing the fabrication temperature also resulted in significant improvements in composite strengths. Good-quality, well-consolidated composites were fabricated at temperatures well below those currently used for SiC/Ti composite fabrication.					
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